

COPY

Form PTO-1390 (Rev. 12-29-99) <b>TRANSMITTAL LETTER TO THE UNITED STATES          DESIGNATED/ELECTED OFFICE (DO/EO/US)          CONCERNING A FILING UNDER 35 U.S.C. 371</b>		US DEPARTMENT OF COMMERCE PATENT AND TRADEMARK OFFICE		ATTORNEY'S DOCKET NO. <b>H 3540 PCT/US</b>	
		U.S. APPLICATION NO. (if known see 37 CFR 1.5) <div style="font-size: 1.5em; font-weight: bold; text-align: center;">09/831432</div>			
INTERNATIONAL APPLICATION NO. <b>PCT/EP99/08289</b>		INTERNATIONAL FILING DATE <b>October 30, 1999</b>		PRIORITY DATE CLAIMED <b>November 9, 1998</b>	
TITLE OF INVENTION <b>CLEAR-RINSING AGENTS FOR MACHINE DISHWASHING</b>					
APPLICANT(S) FOR DO/EO/US <b>Rita Koester, Ansgar Behler, Karl-Heinz Schmid and Michael Neuss</b>					
Applicant herewith submits to the United States Designated/Elected Office (EO/DO/US) the following items and other information:					
1.	<input checked="" type="checkbox"/> This is a <b>FIRST</b> submission of items concerning a filing under 35 U.S.C. 371.				
2.	<input type="checkbox"/> This is a <b>SECOND</b> or <b>SUBSEQUENT</b> submission of items concerning a filing under 35 U.S.C. 371.				
3.	<input type="checkbox"/> This express request to begin national examination procedures (35 U.S.C. 371(f)) at any time rather than delay examination until the expiration of the applicable time limit set in 35 U.S.C. 371(b) and PCT Articles 22 and 39 (1).				
4.	<input checked="" type="checkbox"/> A proper Demand for International Preliminary Examination was made by the 19th month from the earliest claimed priority date.				
5.	<input checked="" type="checkbox"/> A copy of the International Application as filed (35 U.S.C. 371(c)(2)). <div style="margin-left: 20px;">           a. <input type="checkbox"/> is transmitted herewith (required only if not transmitted by the International Bureau).            b. <input checked="" type="checkbox"/> has been transmitted by the International Bureau.            c. <input type="checkbox"/> is not required, as the application was filed in the United States Receiving Office (RO/US).         </div>				
6.	<input checked="" type="checkbox"/> A translation of the International Application into English (35 U.S.C. 371(c)(2)).				
7.	<input checked="" type="checkbox"/> Amendments to the claims of the International Application under PCT Article 19 (35 U.S.C. 371(c)(3)) <div style="margin-left: 20px;">           a. <input type="checkbox"/> are transmitted herewith (required only if not transmitted by the International Bureau).            b. <input type="checkbox"/> have been transmitted by the International Bureau.            c. <input type="checkbox"/> have not been made; however, the time limit for making such amendments has NOT expired.            d. <input checked="" type="checkbox"/> have not been made and will not be made.         </div>				
8.	<input type="checkbox"/> A translation of the amendments to the claims under PCT Article 19 (35 U.S.C. 371(c)(3)).				
9.	<input checked="" type="checkbox"/> An oath or declaration of the inventor(s) (35 U.S.C. 371(c)(4)). <b>(UNEXECUTED)</b>				
10.	<input type="checkbox"/> A translation of the annexes to the International Preliminary Examination Report under PCT Article 36 (35 U.S.C. 371(c)(5)).				
<b>Items 11. to 16. below concern other document(s) or information included:</b>					
11.	<input type="checkbox"/> An Information Disclosure Statement under 37 CFR 1.97 and 1.98.				
12.	<input type="checkbox"/> An assignment document for recording. A separate cover sheet in compliance with 37 CFR 3.28 and 3.31 is included.				
13.	<input checked="" type="checkbox"/> A <b>FIRST</b> preliminary amendment <input type="checkbox"/> A <b>SECOND</b> or <b>SUBSEQUENT</b> preliminary amendment.				
14.	<input type="checkbox"/> A substitute specification.				
15.	<input type="checkbox"/> A change of power of attorney and/or address letter.				
16.	<input type="checkbox"/> Other items or information:				
<b>"Express Mail Post Office to Addressee" service Mailing Label Number</b> <b><u>EL541613752US</u></b>					

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PATENT

Docket No. H 3540 PCT/US

**IN THE UNITED STATES PATENT AND TRADEMARK OFFICE**

RE: PCT/EP99/08289  
International Filing Date: October 30, 1999  
Priority Date Claimed: November 9, 1998  
Applicant: Koester et al.  
Title: CLEAR-RINSING AGENTS FOR MACHINE DISHWASHING  
Applicants' Reference: H 3540 PCT/US

**PRELIMINARY AMENDMENT**

Commissioner for Patents  
Box PCT  
Washington, DC 20231

ATTN: DO/EO/US

Prior to the calculation of fees and examination of the above-identified national stage application pursuant to the accompanying submission under 35 U.S.C. §371, please amend the English translation of the International Application submitted herewith, without prejudice, as follows:

**In the Specification:**

Please amend the instant Specification, without prejudice, as follows:

Please delete all text above line 6 of page 1, and replace the deleted matter with the following new section headings and title of the invention:

--TITLE OF THE INVENTION

Machine Dishwashing Rinse Agents  
and Methods of Using the Same

**BACKGROUND OF THE INVENTION--**

At page 3, between lines 5 and 6 thereof, please insert the following new section heading and new paragraph:

**Preliminary Amendment of U.S. National Stage for International Application  
PCT/EP99/08289 filed October 30, 1999**

**--BRIEF SUMMARY OF THE INVENTION**

The present invention relates, in general, to rinse agents for machine dishwashing containing alkoxyated carboxylic acid esters, particularly those obtained by reaction of carboxylic acid esters and alkylene oxides in the presence of calcined hydrotalcites, and to methods of using the alkoxyated carboxylic acid esters for the production of such rinse agents.--

At page 3, line 25 thereof, please insert the following new section heading:

**--DETAILED DESCRIPTION OF THE INVENTION--**

At page 15, between lines 1 and 2, please add the following new paragraph:

--What is claimed is:--.

On a separate, new page 18, following page 17, please add the following new section heading and paragraph containing an Abstract of the Disclosure:

**--ABSTRACT OF THE DISCLOSURE**

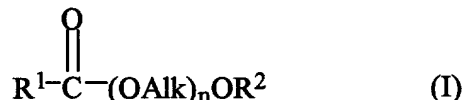
Rinse agents for machine dishwashing which contain alkoxyated carboxylic acid ester(s), and other optional additives, are described. Methods of rinsing tableware surfaces using the described rinsing agents during machine washing are also described.--

**In the Claims:**

Please add new claims 14-36, as follows:

--14. (New) A method of rinsing machine-washed tableware materials, said method comprising:

(a) providing a rinse agent comprising an alkoxyated carboxylic acid ester of the general formula (I):



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wherein  $R^1C(O)$  represents an aliphatic acyl group, each AlkO independently represents an alkoxyate selected from the group consisting of  $CH_2CH_2O$ ,  $CHCH_3CH_2O$  and  $CH_2CHCH_3O$ , n is a number of from 1 to 20, and  $R^2$  represents an aliphatic alkyl group; and

(b) contacting a tableware material surface with the rinse agent during machine washing of the tableware material surface.--

--15. (New) The method according to claim 14, wherein  $R^1C(O)$  represents an aliphatic acyl group having from 8 to 18 carbon atoms, each AlkO represents a  $CH_2CH_2O$ ,  $R^2$  represents a methyl group, and n is a number of from 5 to 15.--

--16. (New) The method according to claim 14, wherein the alkoxyated carboxylic acid ester is prepared by reacting a carboxylic acid and an alkylene oxide in the presence of calcined hydrotalcite.--

--17. (New) The method according to claim 14, wherein the alkoxyated carboxylic acid ester is present in the rinse agent in an amount of from 0.5 to 40% by weight.--

--18. (New) The method according to claim 14, wherein the rinse agent further comprises an additional nonionic surfactant selected from the group consisting of fatty alcohol polyglycol ethers, alk(en)yl oligoglycosides, fatty acid-N-alkyl glucamides, hydroxy mixed ethers, mixed ethers, and mixtures thereof.--

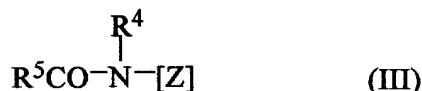
--19. (New) The method according to claim 14, wherein the rinse agent further comprises an alk(en)yl oligoglycoside of the general formula (II):



wherein  $R^3$  represents an alkyl or alkenyl group having from 4 to 22 carbon atoms, each G independently represents a sugar unit containing 5 or 6 carbon atoms and p represents a number of from 1 to 10.--

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--20. (New) The method according to claim 14, wherein the rinse agent further comprises a fatty acid-N-alkyl polyhydroxy alkylamide of the general formula (III):



wherein  $\text{R}^5\text{CO}$  represents an aliphatic acyl group having from 6 to 22 carbon atoms,  $\text{R}^4$  represents an alkyl or hydroxyalkyl group having from 1 to 4 carbon atoms, and  $[\text{Z}]$  represents a linear or branched polyhydroxyalkyl group having from 3 to 12 carbon atoms and from 3 to 10 hydroxyl groups.--

--21. (New) The method according to claim 14, wherein the rinse agent further comprises a fatty alcohol poly(alkylene)glycol ether of the general formula (V):



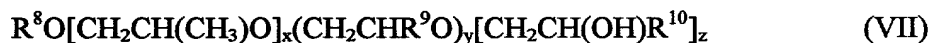
wherein  $\text{R}^6$  represents an alk(en)yl group having from 8 to 22 carbon atoms, each MO independently represents an alkoxide selected from the group consisting of propylene oxide and butylene oxide, p is a number of from 1 to 15 and m is a number of from 0 to 10.--

--22. (New) The method according to claim 14, wherein the rinse agent further comprises a fatty alcohol polyalkylene glycol ether of the general formula (VI):



wherein  $\text{R}^7$  represents an alk(en)yl group having from 8 to 22 carbon atoms, r is a number of from 1 to 10 and q is a number of from 0 to 15.--

--23. (New) The method according to claim 14, wherein the rinse agent further comprises a hydroxy mixed ether of the general formula (VII):



wherein  $\text{R}^8$  represents an alk(en)yl group having from 4 to 18 carbon atoms, each  $\text{R}^9$  independently represents a hydrogen or a methyl or ethyl group, each  $\text{R}^{10}$  independently represents an alkyl group having from 2 to 22 carbon atoms, x is a number of from 0 to 10, y is a number of from 1 to 30 and z is the number 1.--

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--24. (New) The method according to claim 18, wherein the alkoxyated carboxylic acid ester and the additional nonionic surfactant are present in the rinse agent in a ratio by weight of from 10:90 to 80:20.--

--25. (New) The method according to claim 14, wherein the rinse agent further comprises an acid selected from the group consisting of monocarboxylic acids, polycarboxylic acids, and mixtures thereof.--

--26. (New) The method according to claim 25, wherein the acid is present in an amount of from 1 to 50% by weight.--

--27. (New) The method according to claim 17, wherein the rinse agent further comprises an acid selected from the group consisting of monocarboxylic acids, polycarboxylic acids, and mixtures thereof.--

--28. (New) The method according to claim 27, wherein the acid is present in an amount of from 1 to 50% by weight.--

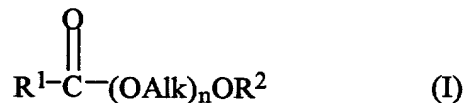
--29. (New) The method according to claim 19, wherein the rinse agent further comprises an acid selected from the group consisting of monocarboxylic acids, polycarboxylic acids, and mixtures thereof.--

--30. (New) The method according to claim 29, wherein the acid is present in an amount of from 1 to 50% by weight.--

--31. (New) A rinsing agent comprising:

(a) an alkoxyated carboxylic acid ester of the general formula (I):

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wherein  $\text{R}^1\text{C}(\text{O})$  represents an aliphatic acyl group, each  $\text{AlkO}$  independently represents an alkoxyate selected from the group consisting of  $\text{CH}_2\text{CH}_2\text{O}$ ,  $\text{CHCH}_3\text{CH}_2\text{O}$  and  $\text{CH}_2\text{CHCH}_3\text{O}$ ,  $n$  is a number of from 1 to 20, and  $\text{R}^2$  represents an aliphatic alkyl group; and

(b) an acid selected from the group consisting of monocarboxylic acids, polycarboxylic acids, and mixtures thereof.--

--32. (New) The rinsing agent according to claim 31, further comprising an additional nonionic surfactant selected from the group consisting of fatty alcohol polyglycol ethers, alk(en)yl oligoglycosides, fatty acid-N-alkyl glucamides, hydroxy mixed ethers, mixed ethers, and mixtures thereof.--

--33. (New) The rinsing agent according to claim 31, further comprising a solubilizer.--

--34. (New) The rinsing agent according to claim 31, wherein the alkoxyated carboxylic acid ester is present in the rinse agent in an amount of from 0.5 to 40% by weight.--

--35. (New) The rinsing agent according to claim 31, wherein the acid is present in an amount of from 1 to 50% by weight.--

--36. (New) The rinsing agent according to claim 32, further comprising a solubilizer, wherein the alkoxyated carboxylic acid ester is present in the rinse agent in an amount of from 0.5 to 40% by weight, and wherein the acid is present in an amount of from 1 to 50% by weight.--

Please cancel claims 1-13, without prejudice.



**Preliminary Amendment of U.S. National Stage for International Application  
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**REMARKS**

Claims 14-36 are currently pending in the instant application.

The Specification has been amended to delete the original section headings and to insert the preferred section headings pursuant to 37 C.F.R. §1.77. A new Title of the Invention has been inserted. An Abstract of the Disclosure, in accordance with the disclosure, has been added. It is submitted that the amendments to the Specification made herein introduce no new matter. All of the amendments to the Specification constitute deletions of original section headings and/or paragraphs, and insertions or additions of new section headings and/or paragraphs. Accordingly, pursuant to 37 C.F.R. §1.121(b)(1)(iii), no separate page captioned "VERSION WITH MARKINGS TO SHOW CHANGES MADE" is necessary. A separate page containing a clean copy of the Abstract of the Disclosure has been added for the Examiner's convenience. Entry of the amendments to the Specification made herein are therefore proper and respectfully requested.

Original claims 1-13 have been canceled and replaced with new claims 14-36 solely for the purpose of improving clarity and grammar, which may suffer in translation, and not for any reason which relates to the statutory requirements for a patent. New claims 14-36 have not been added in response to any rejection, nor in anticipation of any rejection. Applicants respectfully submit that the scope of new claims 14-36 generally corresponds to the scope of original claims 1-13, and that new claims 14-36 are no narrower than original claims 1-13. Furthermore, although a moot point in view of their cancellation, Applicants respectfully submit that original claims 1-13 satisfied the requirements of 35 U.S.C. §112, as filed. New claims 14-36 are supported by the claims as originally filed and in the Specification, for example, at page 3, lines 1-24; at page 4, line 28, through page 5, line 2; and in the Examples. No new matter has been introduced. All of the amendments to the Claims constitute cancellation of original claims and the addition of new claims. Accordingly, pursuant to 37 C.F.R. §1.121(c)(1)(ii), no separate page captioned "VERSION WITH MARKINGS TO SHOW CHANGES MADE" is necessary. Entry is therefore proper and respectfully requested.

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Prompt examination of the instant application in view of the amendments made  
herein is respectfully requested.

Respectfully submitted,

**RITA KOESTER, et al.**

May 9, 2001  
(Date)

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## ABSTRACT OF THE DISCLOSURE

Rinse agents for machine dishwashing which contain alkoxyated carboxylic acid ester(s), and other optional additives, are described. Methods of rinsing tableware surfaces using the described rinsing agents during machine washing are also described.

### Clear-rinsing Agents for Machine Dishwashing

5 This invention relates to rinse agents for machine dishwashing containing alkoxyated carboxylic acid esters, more particularly those obtained by reaction of carboxylic acid esters and alkylene oxides in the presence of calcined hydrotalcites, and to the use of the alkoxyated carboxylic acid esters as a surfactant for the production of the rinse agents.

10 Today, tableware washed by machine often has to meet more stringent requirements than tableware washed by hand. Thus, even dishes completely freed from food remains are evaluated as unsatisfactory when, after machine dishwashing, they still show whitish stains which are attributable to water hardness or other mineral salts and which emanate from dried-on water droplets through lack of wetting agents.

15 Accordingly, in order to obtain sparkling tableware without any stains, rinse agents are now being successfully used. The addition of rinse agents at the end of the wash program ensures that the water drains completely from the tableware so that, at the end of the wash program, the various surfaces are free from residues and sparkling.

20 Commercially available rinse agents are mixtures of nonionic surfactants, solubilizers (for example cumenesulfonate), organic acids (for example citric acid) and solvents (for example ethanol), water and optionally preservatives and perfumes. The function of the surfactants in these rinse agents is to influence the interfacial tension of the water in such a way that it drains from the tableware in a thin coherent film so that no water droplets, streaks or films remain behind after the subsequent drying phase (so-called wetting effect). Another function of the surfactants is to  
25 suppress the foam caused by food residues in the dishwashing machine. Since the rinse agents generally contain acids to improve the clear drying

effect, the surfactants used also have to be relatively stable to hydrolysis by acids.

In addition, only biodegradable, toxicologically and dermatologically safe ingredients are now considered for use in rinse agent formulations.

- 5 Accordingly, the surfactants used in the rinse agents also have to exhibit these properties.

**EP-B1 0 197 434** (Henkel) describes rinse agents which contain mixed ethers as nonionic surfactants. Many different materials (glass, metal, silver, plastic, china) are cleaned in dishwashing machines. This  
10 range of materials has to be thoroughly wetted in the final rinse cycle. Rinse agent formulations containing mixed ethers as sole surfactant component meet these requirements to only a limited extent, if at all, so that the clear rinse or drying effect is unsatisfactory, particularly on plastic surfaces.

15 **DE-A1 19 611 999** and **WO 94/13618** describe alkoxyated carboxylic acid esters which are obtained by homogeneous catalysis in the presence of hydroxides and reducing agents or a co-catalyst. According to both these documents, such compounds may be used in laundry detergents, dishwashing detergents and cleaners.

20 **DE-A-43 26 112** describes low-foaming multipurpose cleaners which contain alkoxyated carboxylic acid esters in admixture with alkyl glycosides and optionally other surfactants, such as alkyl sulfates, alkyl ether sulfates and fatty alcohol polyglycol ethers. These multipurpose cleaners are intended for cleaning hard surfaces, such as clinker bricks, ceramic tiles,  
25 enamels, PVC or wooden floors. In contrast to rinse agents, however, multipurpose cleaners always contain anionic surfactants. In addition, multipurpose cleaners, unlike rinse agents, are intended to generate a rich initial foam. Finally, other surfactant concentrations are used in multipurpose cleaners. Accordingly, multipurpose cleaners and rinse  
30 agents have to meet different requirements.

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acid esters corresponding to formula (I):



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## 25

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acid esters with alkylene oxides using catalysts, more especially calcined hydrotalcite in accordance with **DE-A-39 14 131**, which give compounds with a narrow homolog distribution. According to the invention, alkoxylated carboxylic acid esters corresponding to general formula (I), in which  $R^1CO$  is an aliphatic acyl group containing 6 to 22 carbon atoms, AlkO stands for a  $CH_2CH_2O-$ ,  $CHCH_3CH_2O-$  and/or  $CH_2-CHCH_3O$  group, n has an average value of 3 to 20 and  $R^2$  is an aliphatic alkyl group containing 1 to 22 carbon atoms, are preferred.

Preferred acyl groups are derived from carboxylic acids containing 6 to 22 carbon atoms of natural or synthetic origin, more especially from linear, saturated and/or unsaturated fatty acids, including the technical mixtures thereof obtainable by lipolysis from animal and/or vegetable fats and oils, for example from coconut oil, palm kernel oil, palm oil, soya oil, sunflower oil, rapeseed oil, cottonseed oil, fish oil, bovine tallow and lard. Examples of such carboxylic acids are caproic acid, caprylic acid, 2-ethyl hexanoic acid, capric acid, lauric acid, isotridecanoic acid, myristic acid, palmitic acid, palmitoleic acid, stearic acid, isostearic acid, oleic acid, elaidic acid, petroselic acid, linoleic acid, linolenic acid, elaeostearic acid, arachic acid, gadoleic acid, behenic acid and/or erucic acid. More particularly,  $R^1CO$  is a linear, even-numbered acyl group containing 8 to 18 carbon atoms.

Preferred alkyl groups  $R^2$  are derived from primary, aliphatic monohydric alcohols containing 1 to 22 carbon atoms which may be saturated and/or unsaturated. Examples of suitable monoalcohols are methanol, ethanol, propanol, butanol, pentanol and the hydrogenation products of the above-mentioned carboxylic acids containing 6 to 22 carbon atoms. More particularly,  $R^2$  is a methyl group.

AlkO preferably stands for a  $CH_2CH_2O$  group.

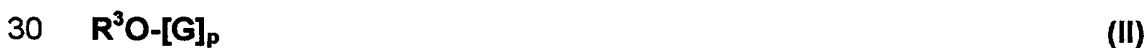
Alkoxylated carboxylic acid esters of formula (I), in which  $R^1CO$  is a linear, even-numbered acyl group containing 8 to 18 carbon atoms, AlkO

stands for a  $\text{CH}_2\text{CH}_2\text{O}$  group,  $n$  has an average value of 5 to 15 and  $\text{R}^2$  is a methyl group, are particularly suitable. Examples of such compounds are carboxylic acid methyl esters alkoxylated with, on average, 5, 7, 9 or 11 moles of ethylene oxide.

- 5 If particularly low-viscosity rinse agents are required, it is advisable to use alkoxylated carboxylic acid esters derived from short-chain carboxylic acids, more particularly those containing 8 to 10 carbon atoms. High cleaning performances are obtained with alkoxylated carboxylic acid esters derived from relatively long-chain carboxylic acids, more particularly
- 10 those containing 12 to 18 carbon atoms.

- The alkoxylated carboxylic acid esters may be present as sole nonionic surfactant in the rinse agents, preferably in quantities of 0.5 to 40% by weight and more particularly in quantities of 5 to 35% by weight. However, the alkoxylated carboxylic acid esters are preferably present in
- 15 the rinse agents in admixture with other nonionic surfactants. Typical examples of suitable other nonionic surfactants are mixed ethers, hydroxy mixed ethers, fatty alcohol polyglycol ethers, alkyl phenol polyglycol ethers, fatty acid amide polyglycol ethers, fatty amine polyglycol ethers, alkoxylated triglycerides, alk(en)yl oligoglycosides, fatty acid-N-alkyl
- 20 glucamides, protein hydrolyzates (more particularly wheat-based vegetable products), polyol fatty acid esters, sugar esters, sorbitan esters and polysorbates. Where the nonionic surfactants contain polyglycol ether chains, they may have a conventional homolog distribution although they preferably have a narrow homolog distribution. Preferred other nonionic
- 25 surfactants are fatty alcohol polyglycol ethers, alkyl oligoglucosides, fatty acid-N-alkyl glucamides, hydroxy mixed ethers and/or mixed ethers.

In a preferred embodiment of the invention, the nonionic surfactants used are alkyl and alkenyl oligoglycosides corresponding to formula (II):





in which  $R^3$  is an alkyl and/or alkenyl group containing 4 to 22 carbon atoms, G is a sugar unit containing 5 or 6 carbon atoms and p is a number of 1 to 10. They may be obtained by the relevant methods of preparative organic chemistry.

The alkyl and/or alkenyl oligoglycosides may be derived from aldoses or ketoses containing 5 or 6 carbon atoms, preferably glucose. Accordingly, the preferred alkyl and/or alkenyl oligoglycosides are alkyl and/or alkenyl oligoglucosides. The index p in general formula (II) indicates the degree of oligomerization (DP), i.e. the distribution of mono- and oligoglycosides, and is a number of 1 to 10. Whereas p in a given compound must always be an integer and, above all, may assume a value of 1 to 6, the value p for a certain alkyl oligoglycoside is an analytically determined calculated quantity which is generally a broken number. Alkyl and/or alkenyl oligoglycosides having an average degree of oligomerization p of 1.1 to 3.0 are preferably used. Alkyl and/or alkenyl oligoglycosides having a degree of oligomerization of less than 1.7 and, more particularly, between 1.2 and 1.4 are preferred from the applicational point of view. The alkyl or alkenyl group  $R^3$  may be derived from primary alcohols containing 4 to 11 and preferably 8 to 10 carbon atoms. Typical examples are butanol, caproic alcohol, caprylic alcohol, capric alcohol and undecyl alcohol and the technical mixtures thereof obtained, for example, in the hydrogenation of technical fatty acid methyl esters or in the hydrogenation of aldehydes from Roelen's oxosynthesis. Alkyl oligoglucosides having a chain length of  $C_8$  to  $C_{10}$  (DP = 1 to 3), which are obtained as first runnings in the separation of technical  $C_{8-18}$  coconut fatty alcohol by distillation and which may contain less than 6% by weight of  $C_{12}$  alcohol as an impurity, and also alkyl oligoglucosides based on technical  $C_{9/11}$  oxoalcohols (DP = 1 to 3) are preferred. In addition, the alkyl or alkenyl group  $R^3$  may also be derived from primary alcohols containing 12 to 22 and preferably 12 to 14 carbon

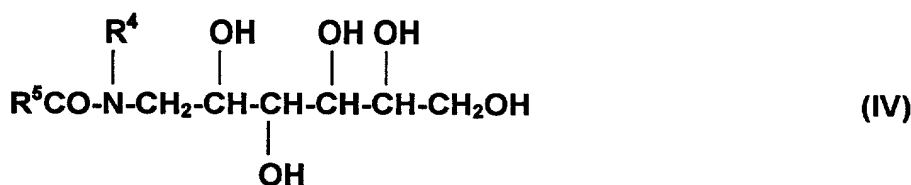
atoms. Typical examples are lauryl alcohol, myristyl alcohol, cetyl alcohol, palmitoleyl alcohol, stearyl alcohol, isostearyl alcohol, oleyl alcohol, elaidyl alcohol, petroselinyl alcohol, arachyl alcohol, gadoleyl alcohol, behenyl alcohol, erucyl alcohol, brassidyl alcohol and technical mixtures thereof  
5 which may be obtained as described above. Alkyl oligoglucosides based on hydrogenated C<sub>12/14</sub> coconut fatty alcohol with a DP of 1 to 3 are preferred.

Another group of preferred other nonionic surfactants are fatty acid-N-alkyl polyhydroxyalkylamides which correspond to formula (III):  
10



15 where R<sup>5</sup>CO is an aliphatic acyl group containing 6 to 22 carbon atoms, R<sup>4</sup> is an alkyl or hydroxyalkyl group containing 1 to 4 carbon atoms and [Z] is a linear or branched polyhydroxyalkyl group containing 3 to 12 carbon atoms and 3 to 10 hydroxyl groups. The fatty acid-N-alkyl polyhydroxyalkylamides are known compounds which may normally be obtained by reductive  
20 amination of a reducing sugar with an alkylamine or an alkanolamine and subsequent acylation with a fatty acid, a fatty acid alkyl ester or a fatty acid chloride. Processes for their production are described in **US 1,985,424**, in **US 2,016,962** and in **US 2,703,798** and in International patent application **WO 92/06984**. An overview of this subject by H. Kelkenberg can be found  
25 in **Tens. Surf. Det.** 25, 8 (1988).

The fatty acid-N-alkyl polyhydroxyalkylamides are preferably derived from reducing sugars containing 5 or 6 carbon atoms, more particularly from glucose. Accordingly, the preferred fatty acid-N-alkyl polyhydroxy-alkylamides are **fatty acid-N-alkyl glucamides** which correspond to  
30 formula (IV):



5

Preferred fatty acid-N-alkyl polyhydroxyalkylamides are glucamides corresponding to formula (IV) in which R<sup>4</sup> is an alkyl group and R<sup>5</sup>CO represents the acyl component of caproic acid, caprylic acid, capric acid, lauric acid, myristic acid, palmitic acid, palmitoleic acid, stearic acid, isostearic acid, oleic acid, elaidic acid, petroselic acid, linoleic acid, linolenic acid, arachic acid, gadoleic acid, behenic acid or erucic acid or technical mixtures thereof. Fatty acid-N-alkyl glucamides (IV) obtained by reductive amination of glucose with methylamine and subsequent acylation with lauric acid or C<sub>12/14</sub> cocofatty acid or a corresponding derivative are particularly preferred. In addition, the polyhydroxyalkylamides may also be derived from maltose and palatinose.

Fatty alcohol polyglycol ethers are particularly preferred as the other nonionic surfactants. The fatty alcohol polyglycol ethers are products of the addition of alkylene oxides containing 2 to 4 carbon atoms (ethylene oxide, propylene oxide and/or butylene oxide) onto fatty alcohols containing 6 to 22 carbon atoms. In one embodiment, the fatty alcohol polyglycol ethers are products of the addition of first ethylene oxide and then optionally propylene oxide and/or butylene oxide onto fatty alcohols of the described type. Within this embodiment, particularly suitable fatty alcohol polyethylene glycol/polypropylene or polybutylene glycol ethers are those corresponding to formula (V):



30

in which R<sup>6</sup> is an alkyl and/or alkylene group containing 8 to 22 carbon

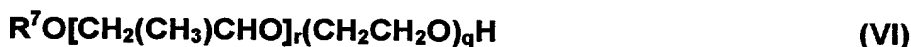
atoms, MO is a propylene oxide and/or a butylene oxide unit, p is a number of 1 to 15 and m is 0 or a number of 1 to 10.

Fatty alcohol polyethylene glycol/polypropylene or polybutylene glycol ethers corresponding to formula (V) may be produced, for example,  
5 in accordance with European patent application **EP-A2-161 537** or **DE-A1 39 28 602** and **DE-A1 39 28 600**.

Particularly suitable representatives are those of formula (V) in which  $R^6$  is an aliphatic, saturated, linear or branched alkyl group containing 8 to 16 carbon atoms, o is a number of 1 to 5 and  $m = 0$ . These  
10 ethers are products of the addition of 1 to 5 moles ethylene oxide onto monohydric alcohols. Suitable monohydric alcohols are the so-called fatty alcohols, such as caproic, caprylic, lauryl, myristyl and stearyl alcohol and the technical mixtures thereof obtained in the high-pressure hydrogenation of technical methyl esters based on fats and oils. Examples of monohydric  
15 branched alcohols are the so-called oxo alcohols which generally carry 2 to 4 methyl groups as branches and are produced by the oxo process and so-called Guerbet alcohols which are branched in the 2-position by an alkyl group. Suitable Guerbet alcohols are 2-ethyl hexanol, 2-butyl octanol, 2-hexyl decanol and/or 2-octyl dodecanol.

20 Other suitable compounds of formula (V) are those in which  $R^6$  is an aliphatic, saturated, linear or branched alkyl group containing 8 to 16 carbon atoms, o is a number of 2 to 7 and m is a number of 3 to 7. These compounds are addition products of monohydric alcohols of the described type alkoxylated first with 2 to 7 moles ethylene oxide and then with 3 to 7  
25 moles propylene and/or butylene oxide.

In another preferred embodiment, the rinse agents contain fatty alcohol polyglycol ethers which are products of the addition of first propylene oxide and then optionally ethylene oxide. Accordingly, the ethers in question are fatty alcohol polypropylene glycol/polyethylene glycol  
30 ethers which preferably correspond to formula (VI):



in which  $\text{R}^7$  is an alkyl and/or alkenyl group containing 8 to 22 carbon atoms,  $r$  is a number of 1 to 10 and  $q$  is a number of 0 to 15.

Compounds such as these are described, for example, in **DE-A1 43 23 252**. Particularly preferred representatives of the compounds corresponding to formula (VI) are those in which  $\text{R}^7$  is an aliphatic, saturated, linear or branched alkyl chain containing 8 to 16 carbon atoms,  $r$  is a number of 1 to 5 and  $q$  is a number of 1 to 6. These compounds are preferably products of the addition of 1 to 5 moles propylene oxide and 1 to 6 moles ethylene oxide onto monohydric alcohols of the type already described.

Particularly preferred other nonionic surfactants also include the so-called mixed ethers. The mixed ethers are products of the addition of ethylene oxide and/or propylene oxide onto fatty alcohols which are end-capped by subsequent reaction with an alkyl chloride in the presence of bases. Particularly suitable mixed ethers are those which have been produced by end-capping with an alkyl halide containing 1 to 8 carbon atoms and more particularly with 1 to 4 carbon atoms of the fatty alcohol polyglycol ethers corresponding to formula (V) and/or (VI). Typical examples are mixed ethers based on a technical  $\text{C}_{12/18}$  or  $\text{C}_{12/14}$  cocoalcohol onto which 5 to 10 moles of ethylene oxide have been added and which have been end-capped with a methyl group or with a butyl group, for example Dehypon® LS-54, LS-104, LT-54, LS-104, LS-531, Henkel KGaA, Düsseldorf/FRG).

Other particularly preferred nonionic surfactants are so-called hydroxy mixed ethers which have been produced by reaction of 1,2-epoxyalkanes with mono-, di- and/or polyhydric alkoxyated alcohols. Preferred hydroxy mixed ethers correspond to formula (VII):



5 in which  $R^8$  is an alkyl and/or alkylene group containing 4 to 18 carbon atoms,  $R^9$  is hydrogen or a methyl or ethyl group,  $R^{10}$  is an alkyl group containing 2 to 22 carbon atoms, x is 0 or a number of 1 to 10, y is a number of 1 to 30 and z is the number 1.

Hydroxy mixed ethers corresponding to formula (VII) are known from the literature and are described, for example, in **WO 96/12001**. They are  
10 produced by reaction of 1,2-epoxyalkanes ( $R^{10}CHOCH_2$ ) with monohydric alkoxyated alcohols. According to the invention, hydroxy mixed ethers derived from alkoxyates of monohydric alcohols ( $z = 1$ ) with the formula  $R^8-OH$  are preferred. Suitable examples of alcohols have already been given in connection with the fatty alcohol polyglycol ethers.

15 The alcohols are used in the form of their alkoxyates which are produced in known manner by reaction of the alcohols with ethylene oxide, propylene oxide and/or butylene oxide. Alkoxyates of alcohols which have been alkoxyated with 10 to 25 moles of ethylene oxide ( $R^9 = \text{hydrogen}$ ,  $x = 0$ ,  $y = 10$  to 25) or with 1 to 3 moles of propylene oxide and then with 10 to  
20 25 moles of ethylene oxide ( $R^9 = \text{hydrogen}$ ,  $x = 1$  to 3,  $y = 10$  to 25) are preferably used.

Most particularly suitable hydroxy mixed ethers corresponding to formula (VII) are those in which  $R^8$  is a saturated linear alkyl chain containing 8 to 14 carbon atoms,  $R^9$  is hydrogen,  $R^{10}$  is a saturated linear  
25 alkyl chain containing 8 to 12 carbon atoms, x is 0 or a number of 1 to 3, y is a number of 10 to 25 and z is the number 1. Hydroxy mixed ethers such as these are described in detail in **DE-A1 37 23 323**.

The rinse agents according to the invention may contain the alkoxyated carboxylic acid esters and the other nonionic surfactants, more  
30 particularly the mixed ethers, hydroxy mixed ethers and the fatty alcohol

polyglycol ethers in a ratio by weight of 10:90 to 80:20 and more particularly 30:70 to 40:60. The ratio by weight of the other nonionic surfactants to one another is less critical.

Besides the surfactants already described, the rinse agent  
5 formulations according to the invention contain mono- and/or polybasic carboxylic acids, preferably hydroxycarboxylic acids, as their most important additives. Typical examples are malic acid (monohydroxy-succinic acid), tartaric acid (dihydroxysuccinic acid), saturated aliphatic dicarboxylic acids, such as oxalic acid, malonic acid, succinic acid, glutaric  
10 acid, adipic acid, gluconic acid (hexanepentahydroxy-1-carboxylic acid), but preferably water-free citric acid. They may be used in the rinse agents in quantities of about 1 to 50% by weight and preferably in quantities of about 5 to 30% by weight. Suitable other additives are, above all, dyes and perfumes and preservatives, preferably in quantities of 0 to 1% by weight.  
15 If desired, the rinse agents may additionally contain solubilizers or hydrotropes. A suitable solubilizer is cumene sulfonate in quantities of 0 to 25% by weight and more particularly in quantities of 0.2 to 15% by weight, expressed as active substance. Recommended hydrotropes are ethanol and/or isopropanol in quantities of 0 to 25% by weight. The balance to  
20 100% by weight is water.

### Commercial Applications

The rinse agents according to the invention contain ecotoxicologically particularly safe ingredients, show excellent wetting  
25 behavior on various materials and excellent foam-suppressing behavior.

The present invention also relates to the use of alkoxyated carboxylic acid esters as a surfactant for the production of rinse agents for machine dishwashing.

The alkoxyated carboxylic acid esters used show excellent solubility  
30 in water without passing through a gel phase. The rinse agents may be

used both in the domestic sector and in the institutional sector.

### Examples

- Rinse agents with the compositions shown in Table 1 (% by weight, based on active substance) were prepared and tested for their clear rinse effect. The clear rinse effect was visually evaluated on the basis of the lime stains observed on glasses, cutlery and plates after they had been cleaned in a commercially available domestic dishwasher at 65°C. The domestic dishwasher was filled with the rinse agents according to the invention and with a comparison formulation (= standard).

If at least 4 out of 5 people visually evaluated a relatively small number of lime stains, the clear rinse effect was judged to be "better than standard". The results are set out in Table 1.

Table 1. Rinse agent formulations

Ingredients	Standard	Ex. 1	Ex. 2	Ex. 3	Ex. 4
$C_{8/18}CO(EO)_{10}CH_3^a$	-	15	2.0	5.0	5.0
Hydroxy mixed ether <sup>b</sup>	-	-	5.0	10.0	-
$C_{12/14}FA+5EO+4PO^c$	15.0	-	8.0	-	10.0
Cumenesulfonate	8.0	5.0	3.0	2.0	5.0
Citric acid	5.0	5.0	5.0	5.0	5.0
	to 100% by weight water				
Clear rinse effect	+	Better than standard	Better than standard	Better than standard	Better than standard

<sup>a</sup>  $C_{8/18}CO(EO)_{10}CH_3$  =  $C_{8/18}$  fatty acid methyl ester ethoxylated with 10 moles ethylene oxide (EO)

<sup>b</sup> Hydroxy mixed ether =  $C_{8/10}$  alcohol cut + 1PO + 22 EO epoxidized with  $\alpha$ -decene epoxide



- c  $C_{12/14}$  FA+5EO+4PO =  $C_{12/14}$  alcohol cut ethoxylated with 5 moles EO and 4 moles propylene oxide (PO)

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**CLAIMS**

1. Rinse agents for machine dishwashing containing alkoxyated carboxylic acid esters corresponding to formula (I):



10 in which  $\text{R}^1\text{CO}$  is an aliphatic acyl group,  $\text{AlkO}$  stands for  $\text{CH}_2\text{CH}_2\text{O}$ ,  $\text{CHCH}_3\text{CH}_2\text{O}$  and/or  $\text{CH}_2\text{CHCH}_3\text{O}$ ,  $n$  is a number of 1 to 20 and  $\text{R}^2$  is an aliphatic alkyl group.

2. Rinse agents as claimed in claim 1, characterized in that they contain alkoxyated carboxylic acid esters corresponding to formula (I) in which  $\text{R}^1\text{CO}$  is an aliphatic acyl group containing 8 to 18 carbon atoms,  
15  $\text{AlkO}$  stands for a  $\text{CH}_2\text{CH}_2\text{O}$  group,  $n$  has an average value of 5 to 15 and  $\text{R}^2$  is a methy group.

3. Rinse agents as claimed in claim 1 or 2, characterized in that they contain alkoxyated carboxylic acid esters corresponding to formula (I) which have been produced by reaction of carboxylic acids with alkylene  
20 oxides in the presence of calcined hydrotalcite.

4. Rinse aids as claimed in any of claims 1 to 3, characterized in that the alkoxyated carboxylic acid esters are present in quantities of 0.5 to 40% by weight.

5. Rinse agents as claimed in any of claims 1 to 4, characterized in that  
25 the alkoxyated carboxylic acid esters are present in admixture with other nonionic surfactants selected in particular from the group of fatty alcohol polyglycol ethers, alkyl oligoglucosides, fatty acid-N-alkyl glucamides, hydroxy mixed ethers and/or mixed ethers.

6. Rinse agents as claimed in claim 5, characterized in that alkoxyated  
30 carboxylic acid esters are present in admixture with alkyl polyglycosides corresponding to formula (II):



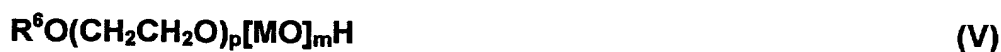
in which  $\text{R}^3$  is an alkyl and/or alkenyl group containing 4 to 22 carbon atoms, G is a sugar unit containing 5 or 6 carbon atoms and p is a number of 1 to 10.

7. Rinse agents as claimed in claim 5, characterized in that alkoxyated carboxylic acid esters are present in admixture with fatty acid-N-alkyl polyhydroxyalkylamides corresponding to formula (III):



where  $\text{R}^5\text{CO}$  is an aliphatic acyl group containing 6 to 22 carbon atoms,  $\text{R}^4$  is an alkyl or hydroxyalkyl group containing 1 to 4 carbon atoms and [Z] is a linear or branched polyhydroxyalkyl group containing 3 to 12 carbon atoms and 3 to 10 hydroxyl groups.

8. Rinse agents as claimed in claim 5, characterized in that alkoxyated carboxylic acid esters are present in admixture with fatty alcohol polyethylene glycol/polypropylene or polybutylene glycol ethers corresponding to formula (V):



in which  $\text{R}^6$  is an alkyl and/or alkylene group containing 8 to 22 carbon atoms, MO is a propylene oxide and/or a butylene oxide unit, p is a number of 1 to 15 and m is 0 or a number of 1 to 10.

9. Rinse agents as claimed in claim 5, characterized in that alkoxyated carboxylic acid esters are present in admixture with fatty alcohol polypropylene glycol/polyethylene glycol ethers corresponding to formula

(VI):



- 5 in which  $\text{R}^7$  is an alkyl and/or alkenyl group containing 8 to 22 carbon atoms,  $r$  is a number of 1 to 10 and  $q$  is a number of 0 to 15.

10. Rinse agents as claimed in claim 5, characterized in that alkoxyated carboxylic acid esters are present in admixture with hydroxy mixed ethers corresponding to formula (VII):

10



- 15 in which  $\text{R}^8$  is an alkyl and/or alkylene group containing 4 to 18 carbon atoms,  $\text{R}^9$  is hydrogen or a methyl or ethyl group,  $\text{R}^{10}$  is an alkyl group containing 2 to 22 carbon atoms,  $x$  is 0 or a number of 1 to 10,  $y$  is a number of 1 to 30 and  $z$  is the number 1.

11. Rinse agents as claimed in any of claims 5 to 10, characterized in that the alkoxyated carboxylic acid esters and the other nonionic surfactants are present in a ratio by weight of 10:90 to 80:20.

- 20 12. Rinse agents as claimed in any of claims 1 to 11, characterized in that they contain mono- and/or polybasic carboxylic acids, preferably citric acid, in quantities of 1 to 50% by weight.

- 25 13. The use of alkoxyated carboxylic acid esters corresponding to formula (I) as a surfactant for the production of rinse agents for machine dishwashing.

Type a plus sign (+) inside this box - ☐0010/PTO  
Rev. 6/95U.S. Department of Commerce  
Patent and Trademark Office**DECLARATION FOR  
UTILITY OR DESIGN  
PATENT APPLICATION**☐ Declaration  
Submitted  
with Initial Filing OR ☒ Declaration  
Submitted after  
Initial FilingAttorney Docket  
Number

H 3540 PCT/US

First Named  
Inventor

KOESTER, Rita

**COMPLETE IF KNOWN**

Application Number

09/831,432

Filing Date

07/30/2001

Group Art Unit

Examiner Name

As a below named inventor, I hereby declare that:

My residence, post office address, and citizenship are as stated below next to my name.

I believe I am the original, first and sole inventor (if only one name is listed below) or an original, first and joint inventor (if plural names are listed below) of the subject matter which is claimed and for which a patent is sought on the invention entitled:

**CLEAR-RINSING AGENTS FOR MACHINE DISHWASHING**

(Title of the Invention)

the specification of which

☐ is attached hereto

OR

☒ was filed on (MM/DD/YYYY) **10/30/1999** as United States Application Number or PCT InternationalApplication Number **PCT/EP99/08289** and was amended on (MM/DD/YYYY) (if applicable).

I hereby state that I have reviewed and understand the contents of the above identified specification, including the claims, as amended by any amendment specifically referred to above.

I acknowledge the duty to disclose information which is material to patentability as defined in Title 37 Code of Federal Regulations, § 1.56.

I hereby claim foreign priority benefits under Title 35, United States Code §119(a)-(d) or §365(b) of any foreign application(s) for patent or inventor's certificate, or §365(a) of any PCT International application which designated at least one country other than the United States of America, listed below and have also identified below, by checking the box, any foreign application for patent or inventor's certificate, or of any PCT International application having a filing date before that of the application on which priority is claimed.

Prior Foreign Application Number(s)	Country	Foreign Filing Date (MM/DD/YYYY)	Priority Not Claimed	Certified Copy Attached? YES NO
198 51 453.0	DE	11/09/1998	<input type="checkbox"/>	<input type="checkbox"/> <input checked="" type="checkbox"/>

☐ Additional foreign application numbers are listed on a supplemental priority sheet attached hereto:

I hereby claim the benefit under Title 35, United States Code §119(e) of any United States provisional application(s) listed below.

Application Number(s)	Filing Date (MM/DD/YYYY)	Additional provisional application numbers are listed on a supplemental priority sheet attached hereto.
		<input type="checkbox"/>

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## DECLARATION

Page 2

I hereby claim the benefit under Title 35, United States Code §120 of any United States application(s), or §365(c) of any PCT international application designating the United States of America, listed below and, insofar as the subject matter of each of the claims of this application is not disclosed in the prior United States or PCT international application in the manner provided by the first paragraph of Title 35, United States Code §112.1 acknowledge the duty to disclose information which is material to patentability as defined in Title 37, Code of Federal Regulations §1.56 which became available between the filing date of the prior application and the national or PCT international filing date of this application.

U.S. Parent Application Number	PCT Parent Number	Parent Filing Date (MM/DD/YYYY)	Parent Patent Number (if applicable)
	PCT/EP99/08289	10/30/1999	

☐ Additional U.S. or PCT international application numbers are listed on a supplemental priority sheet attached hereto.

As a named inventor, I hereby appoint the following attorney(s) and/or agent(s) to prosecute this application and to transact all business in the Patent and Trademark Office connected therewith:

☐ Firm Name  Customer Number  or label

OR

☒ List Attorney(s) and/or agent(s) name and registration number below:

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I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

Name of Sole or First Inventor: ☐ A petition has been filed for this unsigned

Given Name	Rita	Middle Initial		Family Name	Koester	Suffix e.g. Jr.	
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☒ Additional inventors are being named on supplemental sheet(s) attached hereto

## DECLARATION

ADDITIONAL INVENTOR(S)  
Supplemental Sheet

Name of Additional Joint Inventor, if any:

☐ A petition has been filed for this unsigned inventor

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Name of Additional Joint Inventor, if any:

☐ A petition has been filed for this unsigned inventor

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Inventor's Signature	<i>Michael Neuss</i>	Date	<b>05/08/2001</b>
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Name of Additional Joint Inventor, if any:

☐ A petition has been filed for this unsigned inventor

Given Name		Middle Initial		Family Name		Suffix e.g. Jr.	
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Inventor's Signature		Date	
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Residence: City		State		Country		Citizenship	
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☐ Additional inventors are being named on supplemental sheet(s) attached hereto